ADMINISTRATIVE INFORMATION

1. **Project Name:** Project Title **Novel Carbon Films for Next Generation Rotating Equipment Applications**

2. **Lead Organization:** Name University of Illinois at Chicago
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3. **Principal Investigator:** Name Michael McNallan
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4. **Project Partners:** 1. Argonne National Laboratory, subcontractor, Contact: Ali Erdemir, 630-252-6571, erdemir@anl.gov
2. Drexel University, subcontractor, Contact: Yury Gogotsi, 215-895-6446, gogotsi@drexel.edu

5. **Date Project Initiated:** April 1, 2002

6. **Expected Completion Date:** December 31, 2004

PROJECT RATIONALE AND STRATEGY

7. **Project Objective:**

The project aims to combine the unique qualities of two novel carbon technologies to achieve extended wear life and higher energy savings in rotating equipment applications such as, mechanical seals, sliding bearings and shafts. Products to be explored in this project are a super-low friction carbon film developed at Argonne National Laboratory (Near Frictionless Carbon [NFC]) and carbon conversion film with structure and properties ranging from graphite to diamond (Caron Derived Carbon [CDC]) developed at the University of Illinois at Chicago (UIC) and Drexel University.

8. **Technical Barrier(s) Being Addressed:**

Currently, mechanical seals and bearings are produced from hard materials such as SiC or low friction materials, such as graphite. The tribological limitations of these materials, either though frictional losses or wear, limit the performance of the devices. The goal of this project is to develop new carbon based coatings for bearing surfaces with improved frictional and wear properties. The availability of such coatings will produce energy savings through reduced frictional losses in rotating equipment, environmental benefits through reduced fluid leakage and fugitive emissions from production plants, and financial benefits through improved equipment reliability and reduced maintenance costs. The benefits will be cross-cutting and affect all of the Industries of the Future.

9. **Project Pathway:**

Tribological coatings are prepared on SiC seal rings using a high temperature chlorination process for CDC and a plasma CVD process for NFC. The performance of the coatings are tested in the laboratory usng a pin-on-disk tester and physical characterization of the coatings is performed using nanoindentation, microscopy and other techniques. Successful coatings are tested further, by installing coated rings in an instrumented pumping rig to characterize the performance characteristics of the coated rings, such as torque reduction, frictional heating and leakage.

10. **Critical Technical Metrics:** (Please indicate how success or failure will be measured for this project by stating the baseline technical metric(s) and the metric(s) needed for realization of the project objectives.)

Successful coatings will be characterized by the following physical properties
- Uniform continuous coatings
- Raman spectra and physical properties

The following tribological properties
- Friction coefficient less than 0.25 (preferably less than 0.1)
- Minimal wear of coating or counterface pin

And the following pump performance characteristics
- Reduced power requirements
- Frictional heating of less than 260^{o}C in dry running conditions
- No leakage due to seal face wear or spallation

PROJECT PLANS AND PROGRESS

11. **Past Accomplishments:**

- CDC and NFC carbon films have been produced on SiC seal ring material surfaces and characterized using Raman and electron spectroscopy and nanoindentation
- Coated surfaces have been tested in pin on disk apparatus to characterize friction and wear behavior
- Processing conditions for optimal CDC synthesis in terms of high hardness and friction coefficient have been identified
- CDC treated surfaces have been tested in pump seal testers to verify dry run capability (ie frictional heating during high speed operation of a dry pump will not generate temperatures high enough to damage polymers) and long term operation without leakage due to seal face wear
- It was discovered that post treatment of CDC in a high temperature hydrogen containing environment further reduces the friction coefficient.

12. Future Plans

Work will focus on the effects of hydrogen post-treatment on the friction properties of the seal faces with the intention of producing better coatings than have been tested to date.

- CDC films will be synthesized and subjected to hydrogen post-treatment in different gas mixtures, temperatures, and for different times.
- The films will be characterized using optical and electron spectroscopy
- Mechanical properties of the films will be characterized using nano-indentation.

- Tribological properties of the films will be measured in pin-on-disk tests and in simulated pump seal service.

13. Project Changes:

In the course of the work, we learned that it is possible to impart some of the useful characteristics of NFC to CDC by modifying the preparation process for CDC to increase the hydrogen content of the film. The CDC has a high specific surface area, and post-treatment of the CDC in hydrogen after chlorination permits adsorbed chlorine to be replaced by hydrogen. This hydrogenated CDC has lower friction coefficients in tribological couples compared with CDC which has not been subjected to hydrogen post-treatment . Because the hydrogen treatment is also performed at atmospheric pressure and at relatively low temperatures, the hydrogen treated CDC will still be a very low cost material in comparison to films deposited by CVD, PVD, or any plasma enhanced process. Thus, these films may allow us to approach the performance of high cost diamond, DLC, or other carbon coatings at very low cost.

The funding for the project was increased by $223,957 and the performance period was extended from 12 months to 21 months to support this additional activity.

14. **Commercialization Potential, Plans, and Activities:**

The technology developed in this program promises to eliminate many of the problems now experienced on SiC based seals. SiC is a very hard material, however it is very brittle and wears out rapidly during high-speed rotating contacts in face seals. Its friction coefficient is also high, thus it consumes considerable amount of electrical power during operation and the frictional heat generated during unlubricated running can be sufficient to cause combustion of organic materials in contact with the seal. Based on the levels of friction coefficients (i.e., 0.01 to 0.1) measured on new carbon films proposed in this project, it would be possible to improve the energy efficiency of shaft seals in chemical process pumps by several terawatt-hours per year. The particular advantages of the material are for improved lifetime and reliability of pump seals. Pump seals with these advantages would be marketable in the chemical and transportation industries, among other places.

A patent for Carbide Derived Carbon has been issued to the University of Illinois (**Process for Converting a Metal Carbide to Carbon by Etching in Halogens**, M. McNallan, D. Ersoy, Y. Gogotsi, U.S. Patent # 6,579,833 issued June 17, 2003). We have been working with a local producer of pump seals (Chicago-Allis Manufacturing Company, who obtained an option to license the technology from the University of Illinois) to develop it to a commercial scale. Because of some recent decisions by Chicago-Allis, it is likely that the option to license the technology will be transferred to a start-up company, Carbide Derived Technologies, which incorporated late last year. In either case, it is our intention to aggressively pursue the commercialization of these technologies

15. **Patents, Publications, Presentations:** (Please list number and reference, if applicable. If more than 10, please list only 10 most recent.)

Presentations:

- **Structure and Composition Optimization of Carbide-Derived Carbon Films for Tribological Applications,** A. Kovalchenko; Argonne National Laboratory, Y. Gogotsi; Drexel

University, A. Erdemir; Argonne National Laboratory, M. McNallan; University of Illinois at Chicago, B. Carroll; Drexel University, presented at the International Conference on Metallurgical Coatings and Thin Films , San Diego, CA, May 1, 2003.

- **Effect of Annealing and Environment on the Tribological Behavior of Carbide-Derived Carbon Films**, Beth Carroll, Yury Gogotsi, Drexel University, Michael McNallan, University of Illinois at Chicago, Ali Erdemir, Andriy Kovalchenko, Argonne National Laboratory, presented at the Society of Tribologists and Lubrication Engineers 2003 Annual Meeting, New York City, May 1, 2003.
- **Effects of High-Temperature Hydrogenation Treatment on Sliding Friction and Wear Behavior of Carbide-Derived Carbon Films**, by A. Erdemir, A. Kovalchenko, M.J. McNallan, A. Lee, Y. Gogotsi and B. Carroll was presented at the International Conference on Metallurgical Coatings and Thin Films conference in San Diego, CA, April 21, 2004.
- **Effects of Test Conditions and Hydrogen Treatment on Sliding Friction and Wear Behavior of Carbide-Derived Carbon Films**, by M.J. McNallan and A. Lee, A. Erdemir and A. Kovalchenko, and Y. Gogotsi is scheduled for presentation at the Society of Tribologists and Lubrication Engineers 2004 Annual Meeting in Toronto, Canada May 17, 2004.

Thesis:

- The Master of Science thesis: **"Characterization of Carbide Derived Carbon Films on Silicon Carbide for Tribological Applications**," by Beth Carroll was presented and defended at Drexel University, Fall Semester, 2003.

Journal Paper:

- **EFFECT OF HUMIDITY ON THE TRIBOLOGICAL PROPERTIES OF CARBIDE-DERIVED CARBON (CDC) FILMS ON SILICON CARBIDE,** B. Carroll and Y. Gogotsi*, Department of Materials Engineering, Drexel University, A. Kovalchenko and A. Erdemir**,** Energy Technology Division, Argonne National Laboratory, M.J. McNallan, Department of Civil and Materials Engineering, University of Illinois at Chicago, **Tribology Letters, 15**, pp 51-55, July 2003.